Supporting Information for

A bis(pyridyl)-*N*-alkylamine/ Cu(I) catalyst system for aerobic alcohol oxidation

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I. General considerations

^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were recorded on a Bruker 600 MHz Ultrashield Plus spectrometer at 400 MHz and 150 MHz respectively. The chemical shifts (δ) are given in parts per million (ppm) and externally referenced according to the residual protons of the deuterated solvent signal [1]. Coupling constants are reported in Hz. GC analyses were performed on an Agilent 6890 Series GC System with a HP 5 column, 30 m in length, 0.320 mm internal diameter and 0.25 mm film thickness. N_2 served as the carrier gas, acetonitrile (MeCN) and dichloromethane (DCM) were used as the rinsing solutions and cyclohexanone as the internal standard. ESI-MS (positive ion mode) and MALDI-MS spectra were recorded on Bruker microTOF-Q II and Autoflex II spectrometers, by direct injection of the sample or employing DCTB (trans-2-[3-(4-tert-Butylphenyl)-2-methyl-2-propenylidene]malononitrile) as matrix . Aldehyde products were isolated as described below.

II. Optimisation of the catalyst system

We evaluated different bis(pyridyl)-*N*-alkylamine ligands, **L1-L5** (Figure S1), in the oxidation of 1-octanol under previously reported reaction conditions [2]. The following trend in the initial rate was observed: **L1** (0.33 mM·min^{-1}) > **L3** (0.29 mM·min^{-1}) > **L2** (0.27 mM·min^{-1}) > **L4** (0.22 mM·min^{-1}) > **L5** (0.15 mM·min^{-1}).

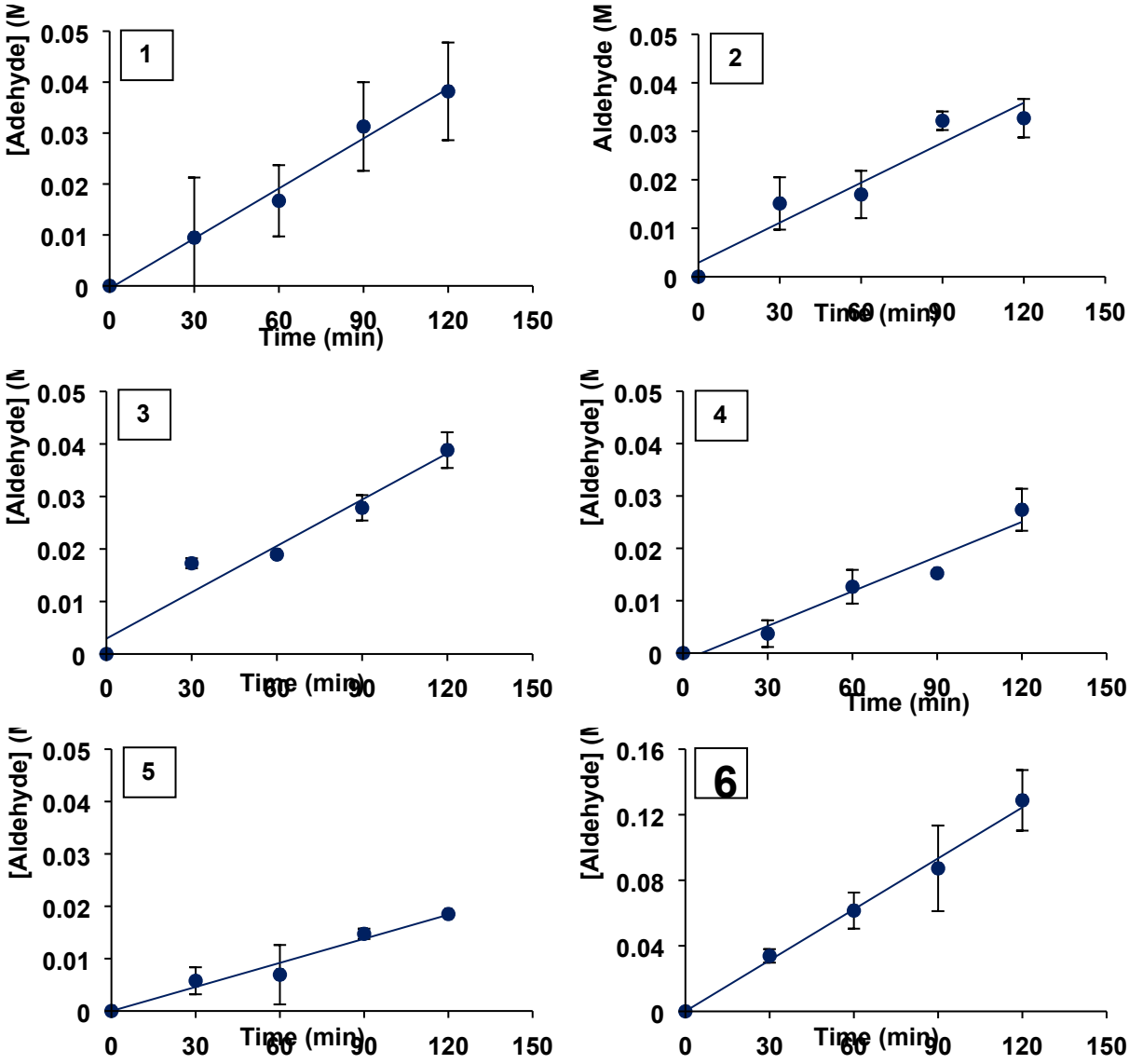


Figure S1: Observed catalytic activity of the different bis(pyridyl)-*N*-alkylamine ligands (**L1-5**) in the oxidation of alcohols. (1) **L1**, 2,2′-dipyridyl-*N*-methylamine; (2) **L2**, 2,2′-dipyridyl-*N*-benzylamine; (3) **L3**, 2,2′-dipyridyl-*N*-methylcyclohexylamine; (4) **L4**, 2,2′-dipyridyl-*N*-neopentylamine; (5) **L5**, (6,6′-dimethyl-2,2'-dipyridyl)-*N*-methylamine and (6) 2,2′-bipyridine (bpy).

Next, we investigated various Cu^{I}- and Cu^{II}-precursors and also different imidazole bases in the aerobic alcohol oxidation of 1-octanol (Table S1 and Figure S2).

Table S1: Evaluation of different Cu^{I}-/Cu^{II}-precursors and imidazole bases in the aerobic alcohol oxidation.[a]

Entry	Ligand	Comment	Time (h)	$k_{initial}$ ($mM \cdot min^{-1}$)[c]	Standard deviation
1	**L3**	$[Cu(MeCN)_4]OTf$	2.5	0.294	0.042
2	**L3**	CuBr	3.0	0.177	0.078
3	**L3**	CuCl	3.0	0.123	0.022
4	**L3**	$CuOTf_2$	2.5	0.015	0.053
5	**L3**	$CuOAc_2 \cdot 2H_2O$	2.5	0.038	0.016
6[b]	**L3**	*N*-methylimidazole	2.5	0.294	0.042
7[b]	**L3**	*N*-tert-butylimidazole	2.5	0.366	0.040
8[b]	**L3**	1-acetylimidazole	2.5	0.043	0.094

[a] alcohol (1.0 mmol, 0.2 M in MeCN), $Cu(MeCN)_4OTf$ (5 mol %, 0.01 M in MeCN), **L** (5 mol %, 0.01 M in MeCN), TEMPO (5 mol %, 0.01 M in MeCN), NMI (10 mol %, 0.02 M in MeCN), 30 °C ± 2 °C.

[b] The same base conditions, as described above in (a), were used during the evaluation of various bases.

[c] The oxidation reactions were done in duplicate to determine an average initial rate value for each reaction.

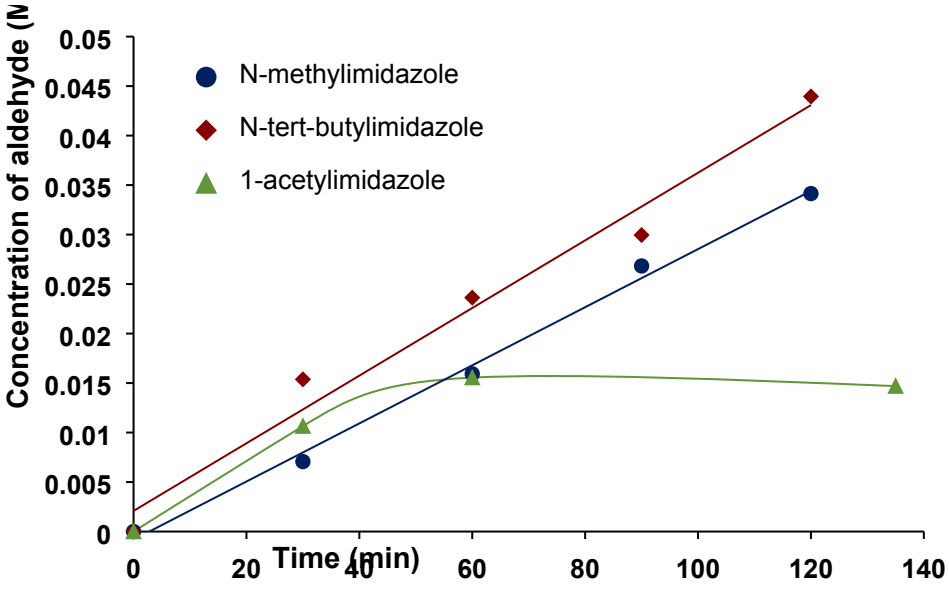


Figure S2: Initial rate data obtained for various *N*-substituted imidazole bases in the aerobic alcohol oxidation of 1-octanol to 1-octanal.

III. Kinetic studies

With the optimised conditions in hand, we conducted a kinetic assessment of our catalyst system under synthetically relevant conditions i.e. under ambient air. The initial rate data were generated for each component except [O_2]. The aerobic alcohol oxidation reaction is performed under O_2 limiting conditions.

Varying [1-octanol]: saturation dependence

The rate of oxidation of 1-octanol exhibits a saturation dependence (Figure S3 and Table S2) on the alcohol substrate concentration. Our observation differs from those of Koskinen et al. [3] and Miyazawa et al. [4] where they found second-order dependence at low [alcohol] and first-order dependence on [alcohol] (mediated by $TEMPO^+$ as the reactive intermediate), respectively. The problem is that although there is a high concentration of alcohol present, the catalyst system is limited in [**L3**Cu] and [NMI] which limits the amount of alkoxide species present in the reaction and the system becomes saturated at higher alcohol concentrations.

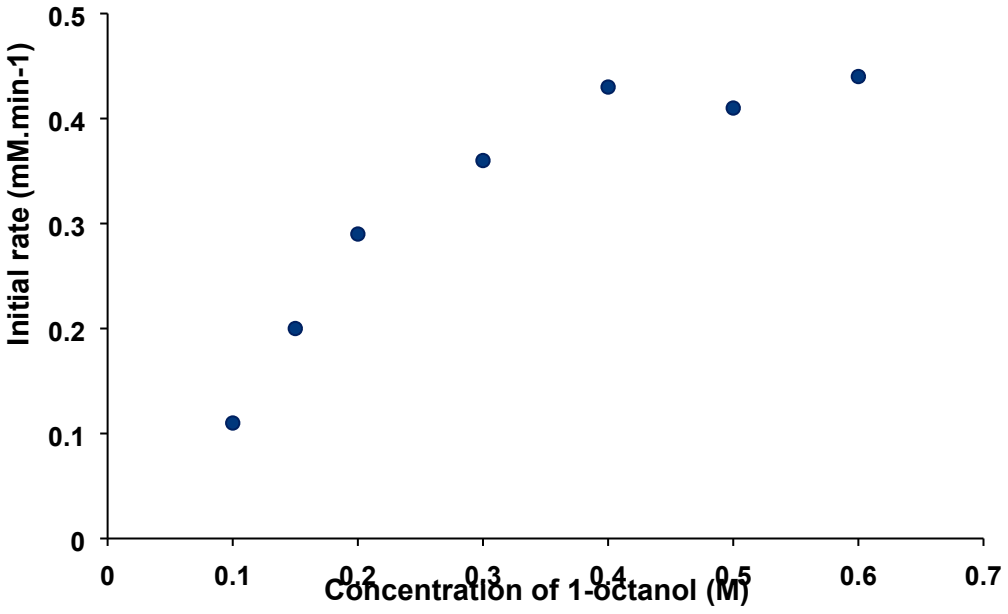


Figure S3: Kinetic data from the oxidation of 1-octanol by (**L3**)$Cu(MeCN)_4OTf$/TEMPO/NMI assessing the kinetic dependence on [1-octanol]. Standard reaction conditions: varying [1-octanol], 10 mM (**L3**)Cu^I, 10 mM TEMPO, 20 mM NMI, 5 mL MeCN, 30 ºC ± 2 ºC, air. The initial rates and standard deviations are summarised in Table S2.

Table S2: Kinetic data from the oxidation of 1-octanol by (**L3**)Cu(MeCN)$_4$OTf/TEMPO/NMI assessing the kinetic dependence on [1-octanol]. Standard reaction conditions: varying [1-octanol], 10 mM (**L3**)Cu^{I}, 10 mM TEMPO, 20 mM NMI, 5 mL MeCN, 30 ºC ± 2 ºC, air.

Concentration (M)	$k_{initial}$ (mM·min^{-1})	Standard deviation
0.1	0.114	0.021
0.15	0.20	0.057
0.2	0.294	0.042
0.3	0.361	0.042
0.4	0.434	0.028
0.5	0.408	0.014
0.6	0.443	0.042

Varying [TEMPO]: saturation dependence

Table S3: Kinetic data from the oxidation of 1-octanol by (**L3**)Cu(MeCN)$_4$OTf/TEMPO assessing the kinetic dependence on [TEMPO]. Standard reaction conditions: 0.2 M 1-octanol, 10 mM (**L3**)Cu^{I}, varying [TEMPO], 20 mM NMI, 5 mL MeCN, 30 ºC ± 2 ºC, air.

Concentration (mM)	$k_{initial}$ (mM·min^{-1})	Standard deviation
1.25	0.014	0.020
2.5	0.123	0.025
5	0.199	0.066
10	0.294	0.041
20	0.467	0.029
30	0.506	0.029
40	0.598	0.061
50	0.571	0.034
60	0.662	0.015

Varying [NMI]: saturation dependence

Table S4: Kinetic data from the oxidation of 1-octanol by (**L3**)Cu(MeCN)$_4$OTf/TEMPO assessing the kinetic dependence on [NMI]. Standard reaction conditions: 0.2 M 1-octanol, 10 mM (**L3**)Cu^{I}, 10 mM TEMPO, varying [NMI], 5 mL MeCN, 30 ºC ± 2 ºC, air.

Concentration (mM)	$k_{initial}$ (mM·min^{-1})	Standard deviation
2.5	0.101	0.056
10	0.216	0.020
20	0294	0.041
40	0.436	0.073
50	0.448	0.044

Varying [L3Cu]: saturation dependence

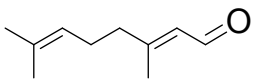
Table S5: Kinetic data from the oxidation of 1-octanol by (**L3**)Cu(MeCN)$_4$OTf/TEMPO assessing the kinetic dependence on [LCu]. Standard reaction conditions: 0.2 M 1-octanol, varying [(**L3**)Cu^{I}], 10 mM TEMPO, 20 mM NMI, 5 mL MeCN, 30 ºC ± 2 ºC, air.

Concentration (mM)	$k_{initial}$ (mM·min^{-1})	Standard deviation
1.25	0.0002	0.036
2.5	0.116	0.016
5	0.212	0.003
10	0.294	0.041
20	0.542	0.003
40	0.610	0.118
50	0.595	0.025

IV. Conversion and characterisation of aldehyde products, Table 2

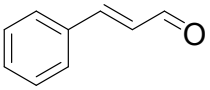
All alcohols were obtained from Sigma-Aldrich and used as received and the converted aldehydes were isolated by column chromatography. The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra data were compared to those previously reported in literature.

Geranial



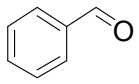
The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 3 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the green oil obtained purified by column chromatography: Hexane/EtOAc (2:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.95, R_f (ligand) = 0.46. The aldehyde was isolated as a lightly coloured oil. Yield = 144 mg, 95 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.99-10.0 (d, $^{3}J_{H-H}$ = 8, 1H *H*C=O), δ 5.88-5.89 (d, *J* = 8, 1H, C=C*H*), δ 5.06-5.08 (m, 1H, C=C*H*), δ 2.19-2.24 (m, 4H, 2CH_2), δ 2.17 (s, 3H, CH_3), δ 1.69 (s, 3H, CH_3), δ 1.61 (s, 3H, CH_3). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$): δ 191.38 (H*C*=O), δ 163.94 (R_2C=C), δ 132.97 (R_2C=C), δ 127.45 (C=*C*), δ 122.59 (C=*C*(H)R), δ 40.65 (CH_2), δ 25.67 (CH_2), δ 25.70 (CH_2), δ 17.77 (CH_3), δ 17.63 (CH_3). Spectral properties are consistent with literature values [5].

Cinnamaldehyde



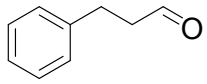
The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 2 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the red oil obtained purified by column chromatography: Hexane/EtOAc (2:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.72, R_f (ligand) = 0.27. The aldehyde was isolated as a pale-yellow oil. Yield = 129 mg, 98 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.64-9.65 (d, 1H, $^{3}J_{H-H}$ = 7.53 *H*C=O); δ 7.50-7.51 (m, 2H, H_{Ar} and C=C*H*); δ 7.37-7.42 (m, 4H, H_{Ar}); δ 6.64-6.68 (dd, 1H, $^{3}J_{H-H}$ = 16.15, 7.91, C=C*H*). $^{13}C\{^{1}H\}$ (150 MHz, $CDCl_3$): δ 193.47 (H*C*=O); δ 152.53 ($C_{C=C}$); δ 133.68 (C_{Ar}); δ 130.99 (C_{Ar}); δ 128.96 ($C_{C=C}$); δ 128.81 (C_{Ar}); δ 128.23 (C_{Ar}). Spectral features are consistent with those reported previously [2].

Benzaldehyde



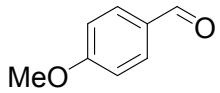
The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 2 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the green oil obtained purified by column chromatography: Hexane/EtOAc (3:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.87, R_f (ligand) = 0.40. The aldehyde was isolated as a pale-orange oil. Yield = 97 mg, 92 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.04 (s, 1H, *H*C=O); δ 7.90-7.91 (d, 2H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}); δ 7.64-7.67 (t, 1H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}); δ 7.54-7.56 (t, 2H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}). ^{13}C{^{1}H} (150 MHz, $CDCl_3$):δ 192.23 (H*C*=O), δ 136.22 (C_{Ar}); δ 134.30 (C_{Ar}); δ 129.58 (C_{Ar}); δ 128.84 (C_{Ar}).Spectral features are consistent with those reported previously [5].

3-phenylpropanal



The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 7 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the green oil obtained purified by column chromatography: Hexane/EtOAc (9:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.40, R_f (ligand) = 0.17. The aldehyde was isolated as a colourless oil. Yield: 127 mg, 95 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.82 (s, 1H, *H*C=O); δ 7.31-7.34 (t, 2H, $^3J_{H\text{-}H}$ = 7.45, H_{Ar}); δ 7.22-7.24 (m, 3H, H_{Ar}); δ 2.97-2.99 (t, 2H, $^3J_{H\text{-}H}$ = 7.45, CH_2); δ 2.78-2.81 (t, 2H $^3J_{H\text{-}H}$ = 7.45, CH_2). ^{13}C{^{1}H} (150 MHz, $CDCl_3$): δ 201.54 (H*C*=O), δ 140.24 (C_{Ar}); δ 128.49 (C_{Ar}); δ 128.19 (C_{Ar}); δ 126.18 (C_{Ar}); δ 46.14 (*C*H_2); δ 27.98 (*C*H_2). Spectral features are consistent with those reported previously [6].

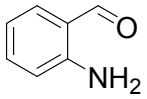
4-methoxybenzaldehyde



The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 2.5 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the red oil obtained purified by column chromatography: Hexane/EtOAc (3:1) as eluent using 5 g Silica

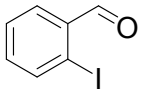
gel. R_f (aldehyde) = 0.38, R_f (ligand) = 0.21. The aldehyde was isolated as a pale-yellow oil. Yield = 225 mg, 90 %.^{1}H NMR (400 MHz, $CDCl_3$): δ 9.86 (s, 1H, *H*C=O); δ 7.81-7.82 (d, 2H, $^3J_{H\text{-}H}$ = 8.66, H_{Ar}); δ 6.98-6.99 (d, 2H, $^3J_{H\text{-}H}$ = 8.66, H_{Ar}); δ 3.86 (s, 3H, CH_3). $^{13}C\{^1H\}$ (150 MHz, $CDCl_3$): δ 190.20 (H*C*=O), δ 163.99 (C_{Ar}); δ 131.37 (C_{Ar}); δ 129.30 (C_{Ar}); δ 113.71 (C_{Ar}); δ 55.01 (C_{Me}). Spectral features are consistent with those reported previously [5].

2-aminobenzaldehyde



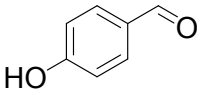
The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 3 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the red oil obtained purified by column chromatography: Hexane/EtOAc (3:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.56, R_f (ligand) = 0.39. The aldehyde was isolated as a yellow oil. Yield = 109 mg, 90 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.85 (s, 1H, *H*C=O); δ 7.46-7.47 (d, 1H, $^3J_{H\text{-}H}$ = 7.91, H_{Ar}); δ 7.28-7.31 (t, 1H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}); δ 6.72-76.74 (t, 1H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}); δ 6.63-6.64 (d, 1H, $^3J_{H\text{-}H}$ = 8.28, H_{Ar}); δ 6.13 (br. s, 2H, NH_2). $^{13}C\{^1H\}$ (150 MHz, $CDCl_3$): δ 193.97 (H*C*=O), δ 149.79 (C_{Ar}); δ 135.61 (C_{Ar}); δ 135.09 (C_{Ar}); δ 118.67 (C_{Ar}); δ 116.23 (C_{Ar}); δ 115.91 (C_{Ar}). Spectral features are consistent with those reported previously [7].

2-iodobenzaldehyde



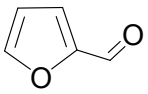
The reaction was performed in a conventional test tube open to air. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 3 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the red oil obtained purified by column chromatography: Hexane/EtOAc (3:1) as eluent using 5 g Silica gel. R_f (aldehyde) = 0.48, R_f (ligand) = 0.29. The aldehyde was isolated as a pale-yellow oil. Yield = 225 mg, 97 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.08 (s, 1H, *H*C=O); δ 7.96-7.97 (d, 1H, $^3J_{H\text{-}H}$ = 7.53, H_{Ar}); δ 7.89-7.90 (d, 1H, $^3J_{H\text{-}H}$ = 7.53, H_{Ar}); δ 7.47-7.49 (t, 1H, $^3J_{H\text{-}H}$ = 7.53, H_{Ar}); δ 7.29-7.32 (t, 1H, $^3J_{H\text{-}H}$ = 7.15, H_{Ar}). $^{13}C\{^1H\}$ (150 MHz, $CDCl_3$): δ 194.45 (H*C*=O), δ 139.42 (C_{Ar}); δ 134.32 (C_{Ar}); δ 129.01 (C_{Ar}); δ 127.63 (C_{Ar}); δ 99.71 (C_{Ar}). Spectral features are consistent with those reported previously [8].

4-hydroxybenzaldehyde



The reaction was performed in a conventional test tube open to air, employing DMF:MeCN (2 ml: 3 ml) as reaction solvent. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 23 h of reaction. After the allotted time the reaction solvent was removed *in vacuo* and the green oily residue was taken up in Et_2O (10 ml) and extracted with water (3 x 50 ml) portions. The organic layer was separated, dried over $MgSO_4$ and the solvent removed. R_f (aldehyde) = 0.54, R_f (ligand) = 0.27. The pale yellow-brown solid (aldehyde) was washed with hexane and filtered as a pale yellow solid. Yield = 122 mg, 92 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.86 (s, 1H, *H*C=O); δ 7.81-7.82 (d, 2H, $^3J_{H\text{-}H}$ = 8.83, H_{Ar}); δ 6.97-6.98 (d, 2H, $^3J_{H\text{-}H}$ = 8.83, H_{Ar}); δ 2.02 (br, s, 1H, OH). $^{13}C\{^1H\}$ (150 MHz, $CDCl_3$): δ 191.43 (H*C*=O), δ 161.81 (C_{Ar}); δ 132.71 (C_{Ar}); δ 130.00 (C_{Ar}); δ 116.21 (C_{Ar}). Spectral features are consistent with those reported previously.[9]

Furfural



The reaction was run in a round bottom flask, fitted with a septum, under oxygen. The reaction was monitored by TLC which showed complete conversion to the aldehyde after 3 h (100 % Methanol). After the allotted time the reaction mixture was purified by aqueous workup (Purification Method 2). R_f (aldehyde) = 0.74, R_f (ligand) = 0.33. The aldehyde was isolated as a brown oil. Yield = 32.1 mg, 33 %. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.69 (s, 1 H, *H*C=O), δ 7.72 (m, 1 H), δ 7.28-7.29 (d, 1H, $^3J_{H\text{-}H}$ = 3.56, H_{Ar}), δ 6.63-6.64 (dd, 1H, $^3J_{H\text{-}H}$ = 3.57, 1.92, H_{Ar}). $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): δ 177.997 (H*C*=O), δ 153.05 (C_{Ar}); δ 148.26 (C_{Ar}); δ 121.14 (C_{Ar}); δ 112.72 (C_{Ar}). Spectral features are consistent with those reported previously [5].

V. NMR spectral data of converted aldehyde products

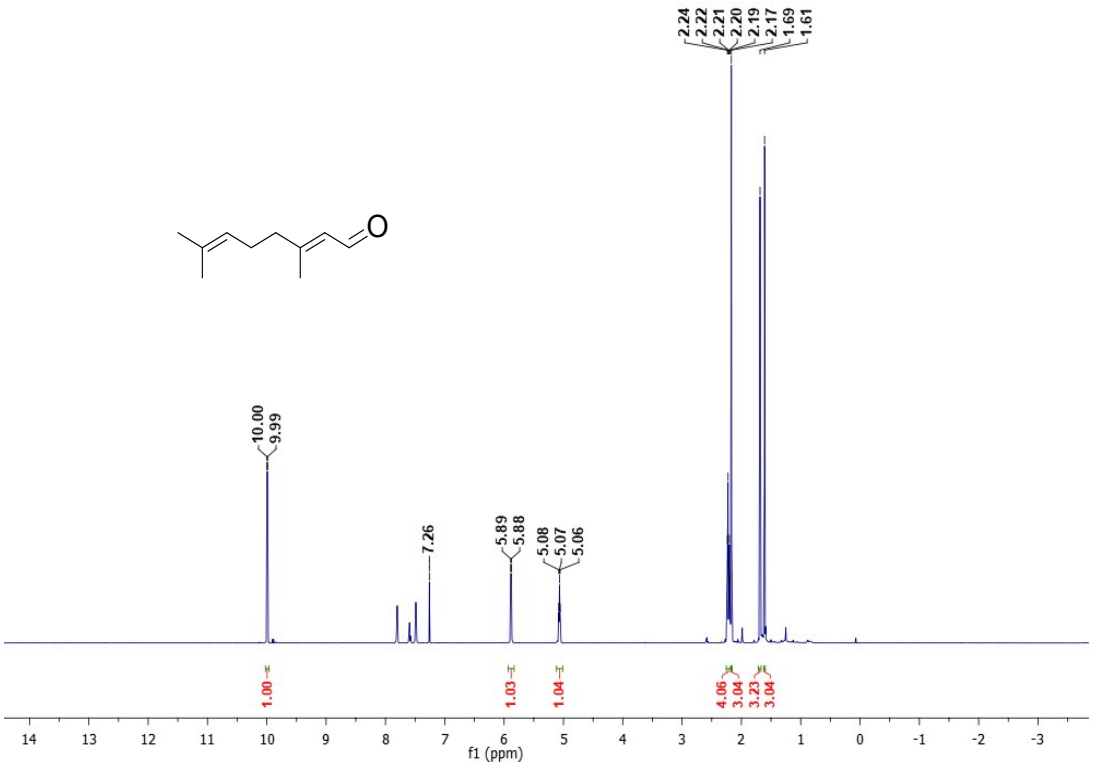


Figure S5: ^{1}H NMR spectrum of **Geranial** in $CDCl_3$.

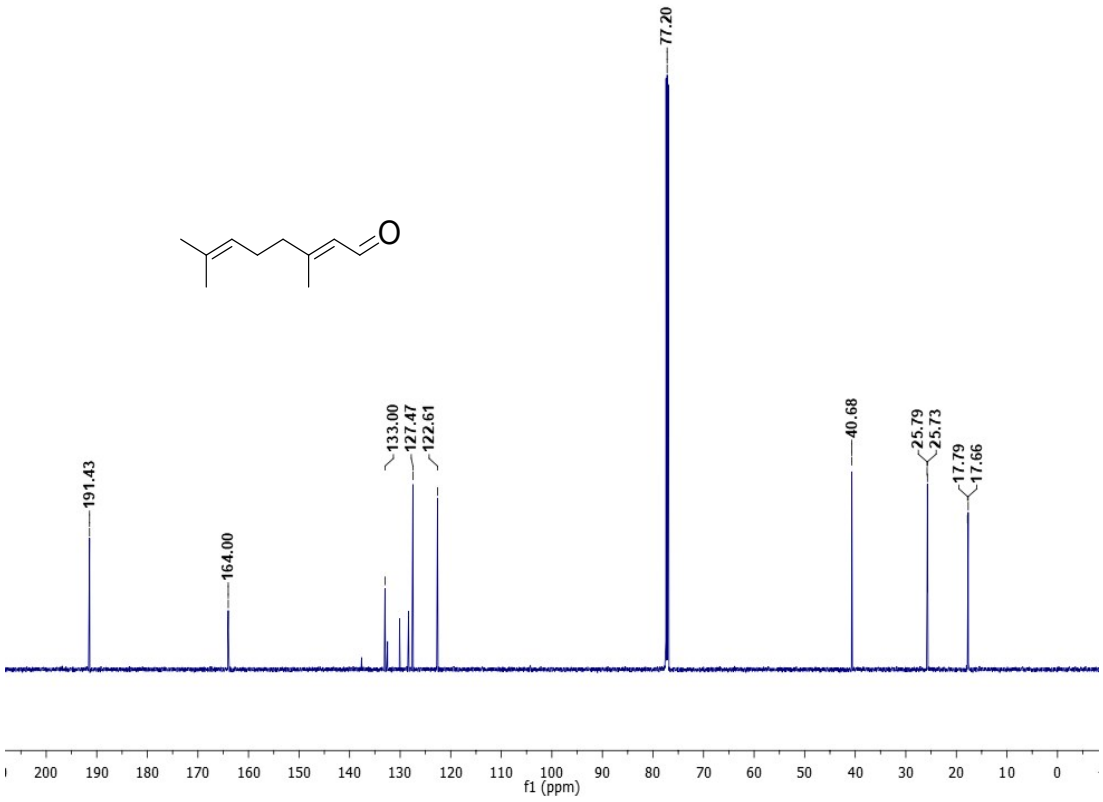


Figure S6: $^{13}C\{^{1}H\}$ NMR spectrum for **Geranial** in $CDCl_3$.

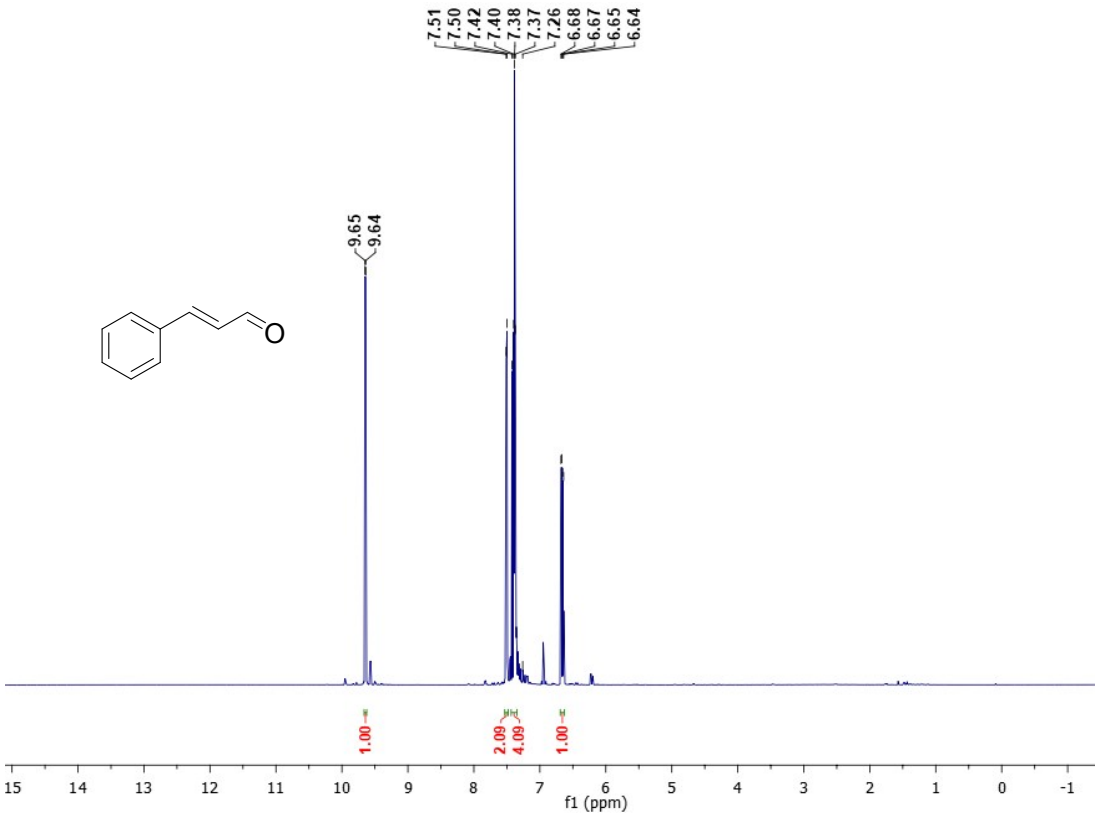


Figure S7: ^{1}H NMR spectrum for **Cinnamaldehyde** in $CDCl_3$.

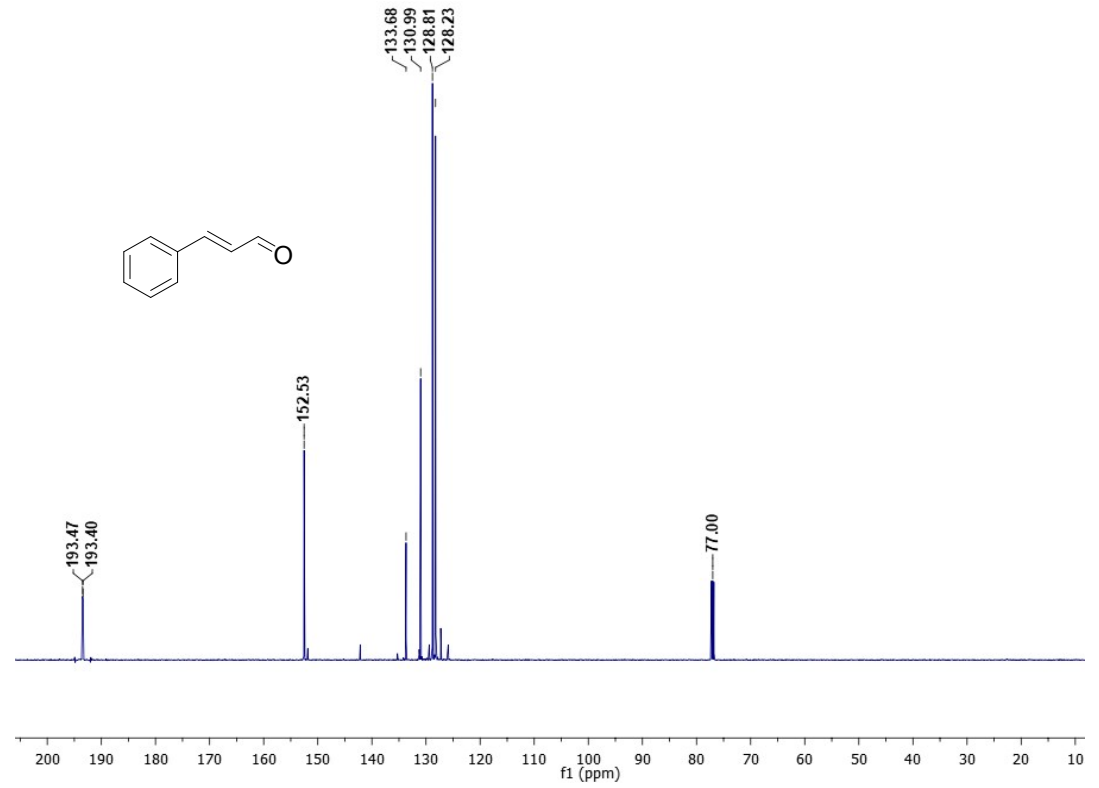


Figure S8: $^{13}C\{^{1}H\}$ NMR spectrum for **Cinnamaldehyde** in $CDCl_3$.

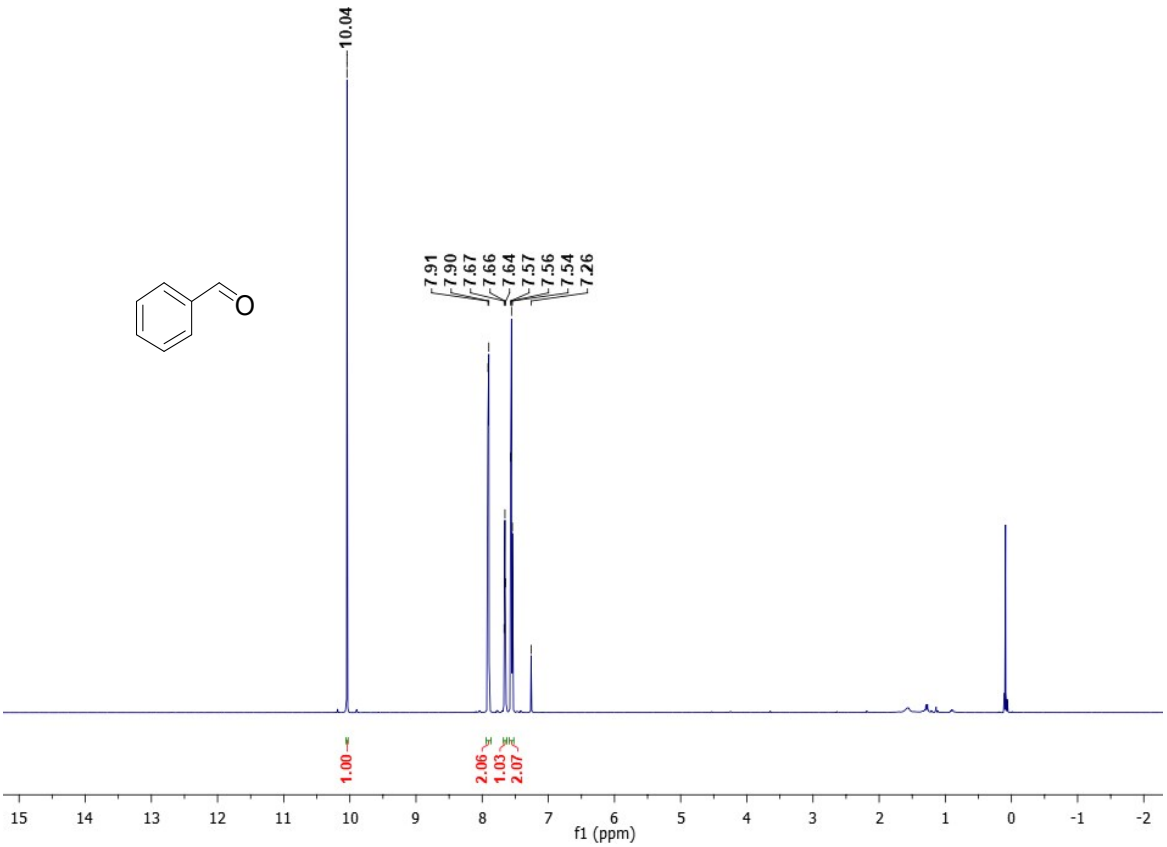


Figure S9: ^{1}H NMR spectrum of **Benzaldehyde** in $CDCl_3$.

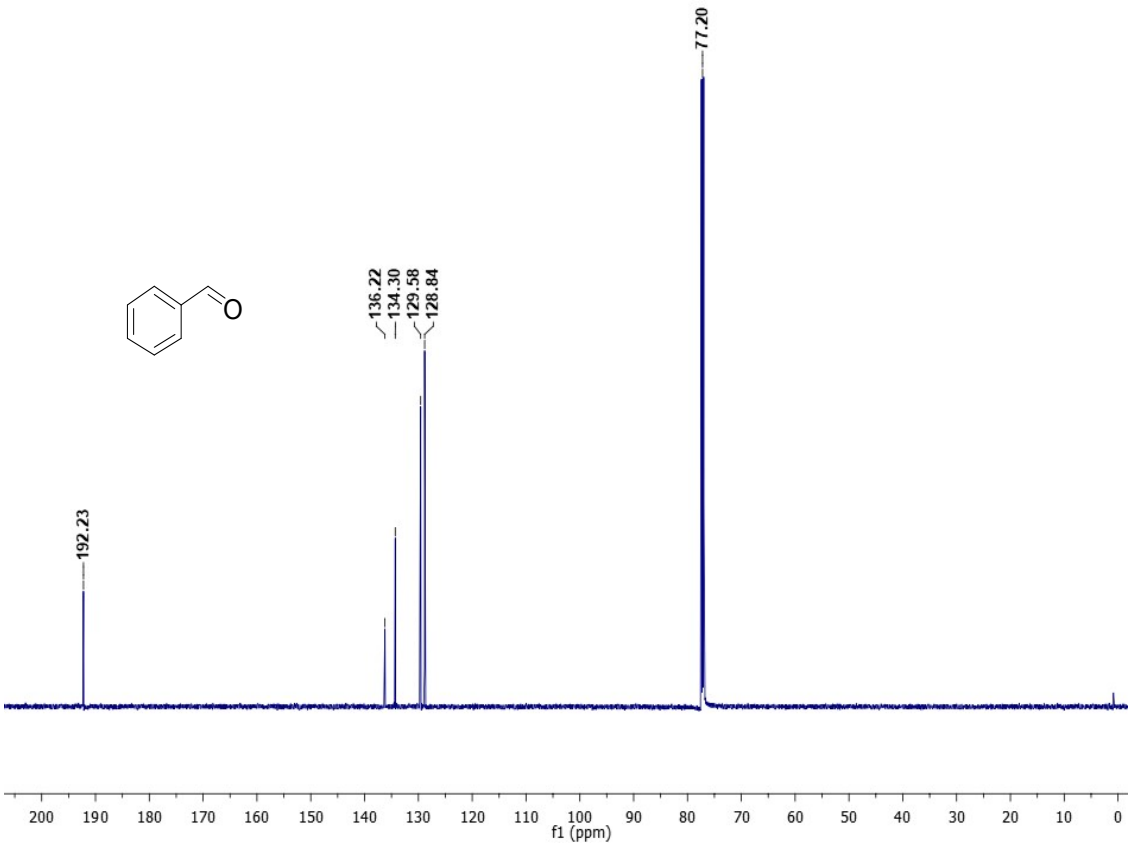


Figure S10: $^{13}C\{^{1}H\}$ NMR spectrum of **Benzaldehyde** in $CDCl_3$.

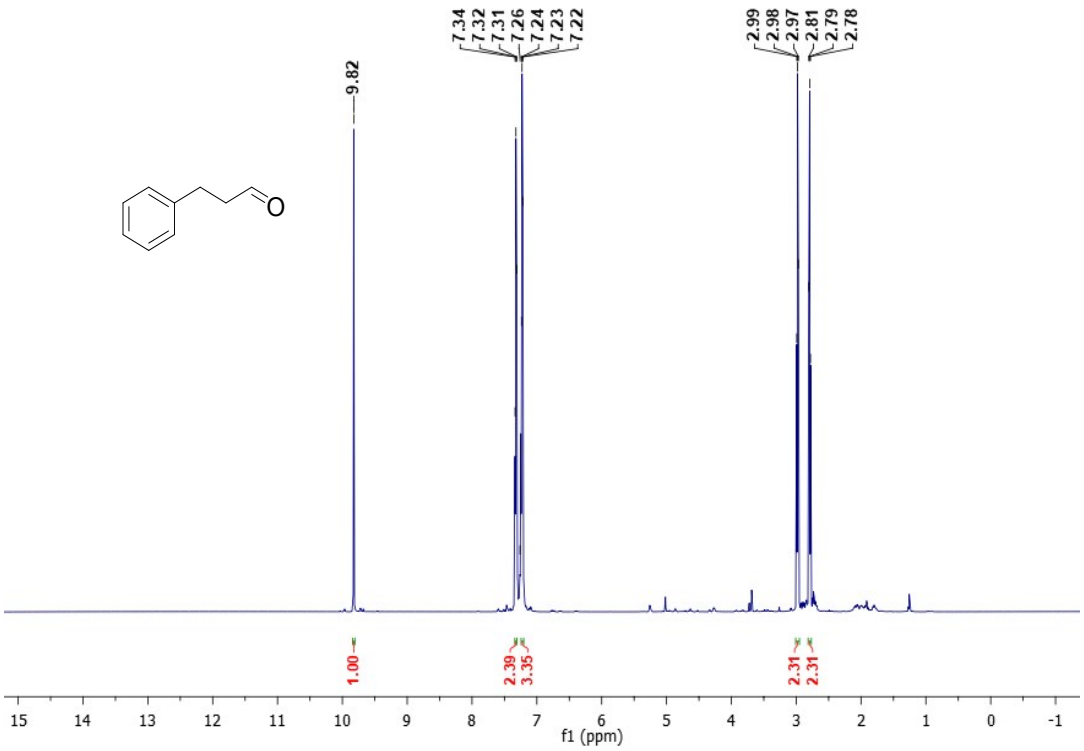


Figure S11: ^{1}H NMR spectrum of **3-phenylpropanol** in $CDCl_3$.

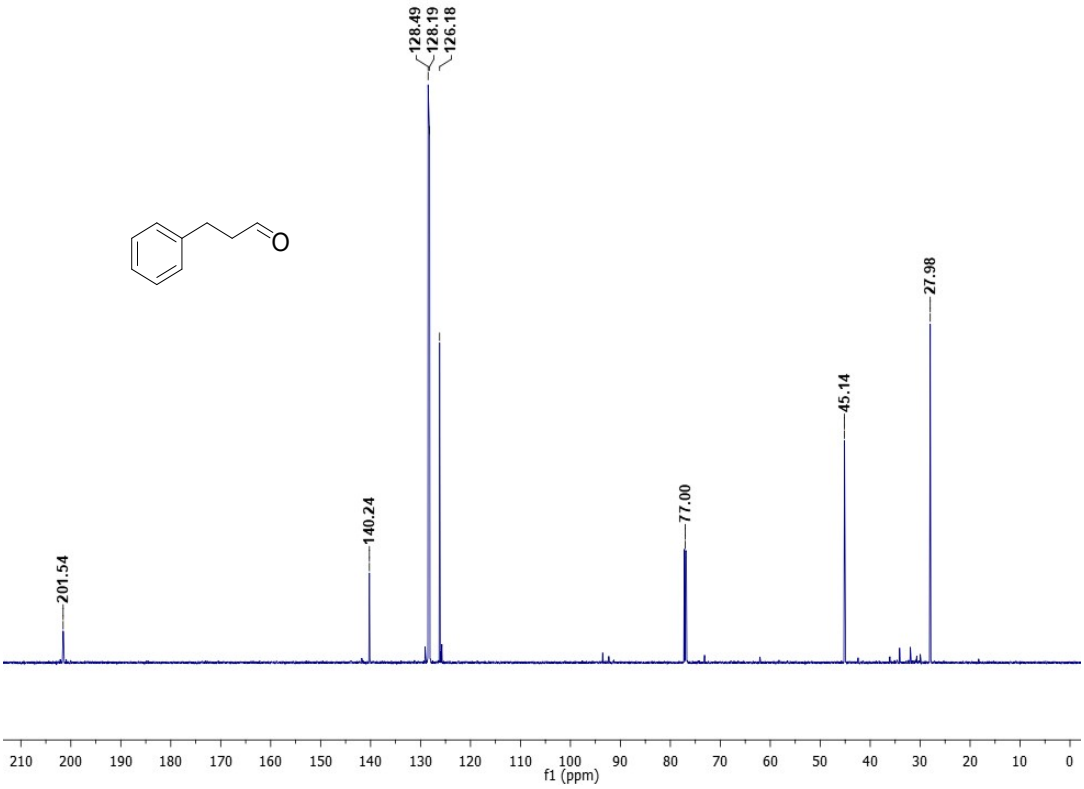


Figure S12: $^{13}C\{^{1}H\}$ NMR spectrum for **3-phenylpropanol** in $CDCl_3$.

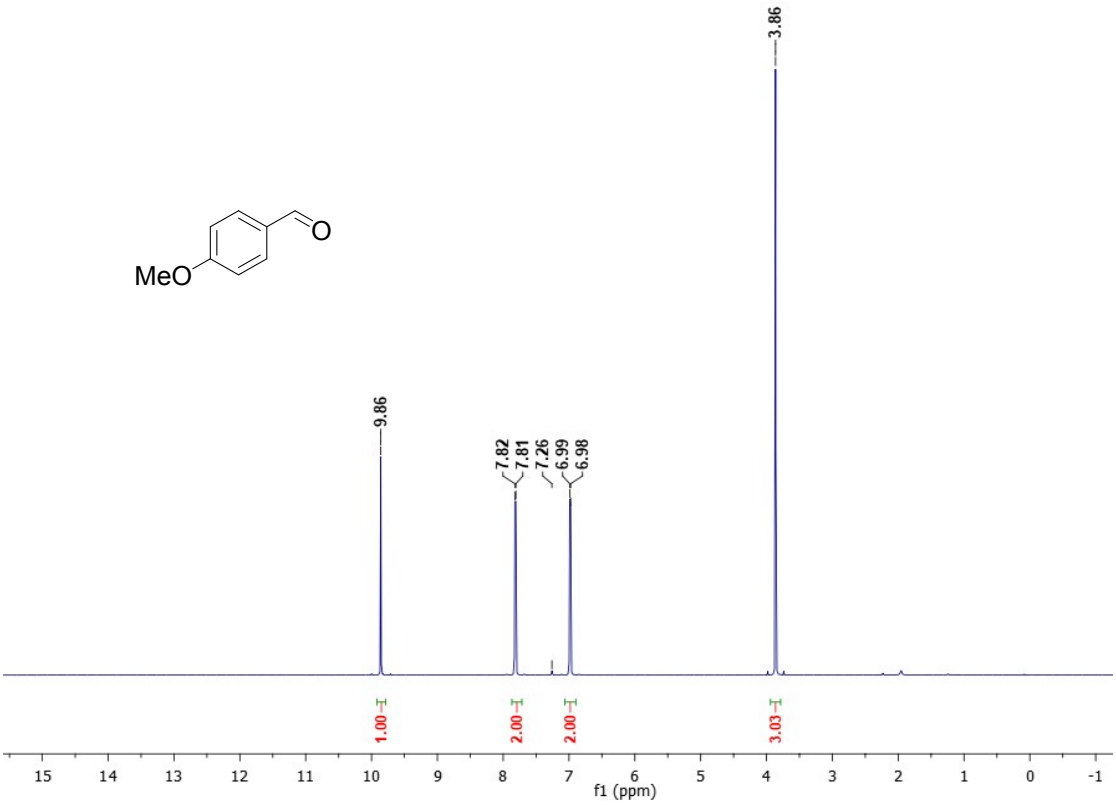


Figure S13: ^{1}H NMR spectrum for **4-methoxybenzaldehyde** in $CDCl_3$.

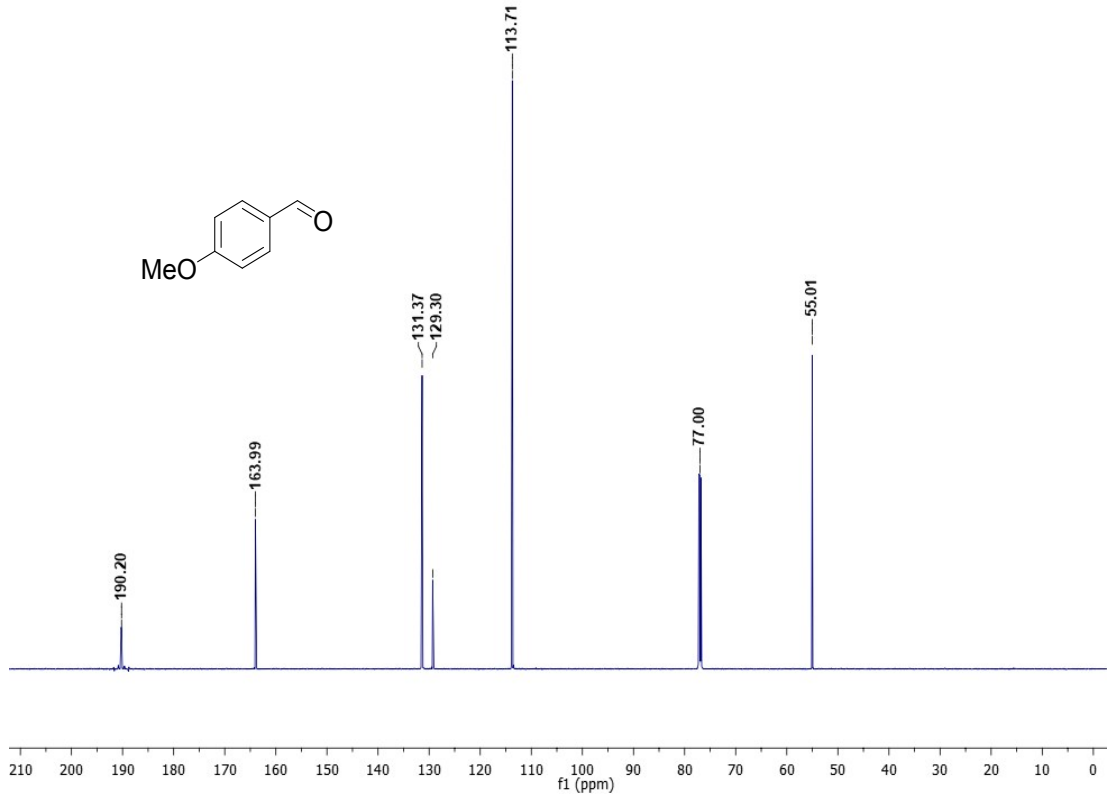


Figure S14: $^{13}C\{^{1}H\}$ NMR spectrum for **4-methoxybenzaldehyde** in $CDCl_3$.

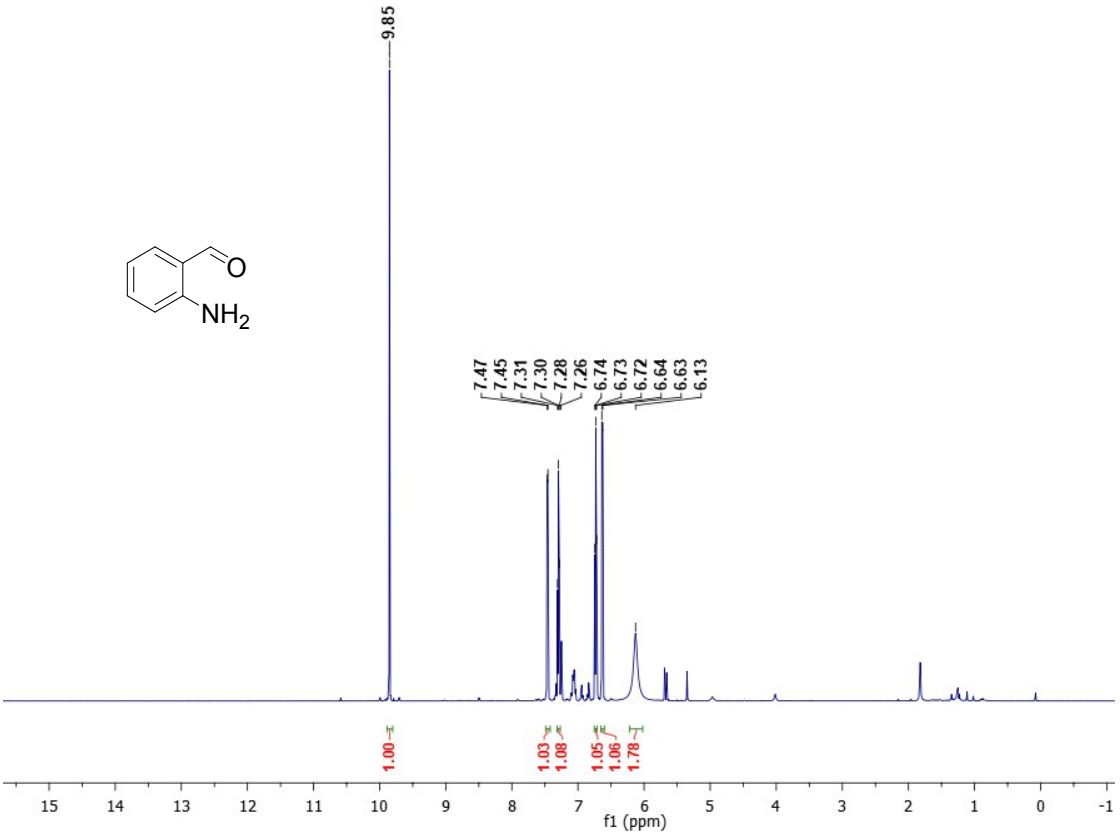


Figure S15: ^{1}H NMR spectrum for **2-aminobenzaldehyde** in $CDCl_3$.

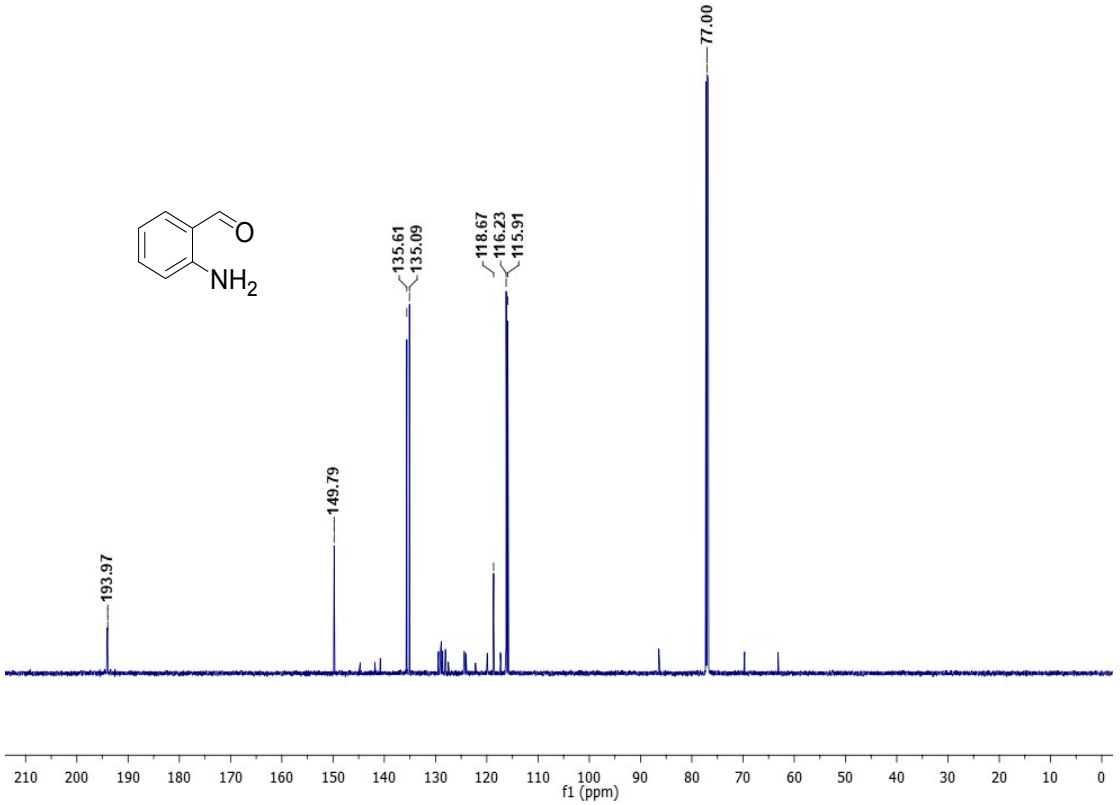


Figure S16: $^{13}C\{^{1}H\}$ NMR spectrum of **2-aminobenzaldehyde** in $CDCl_3$.

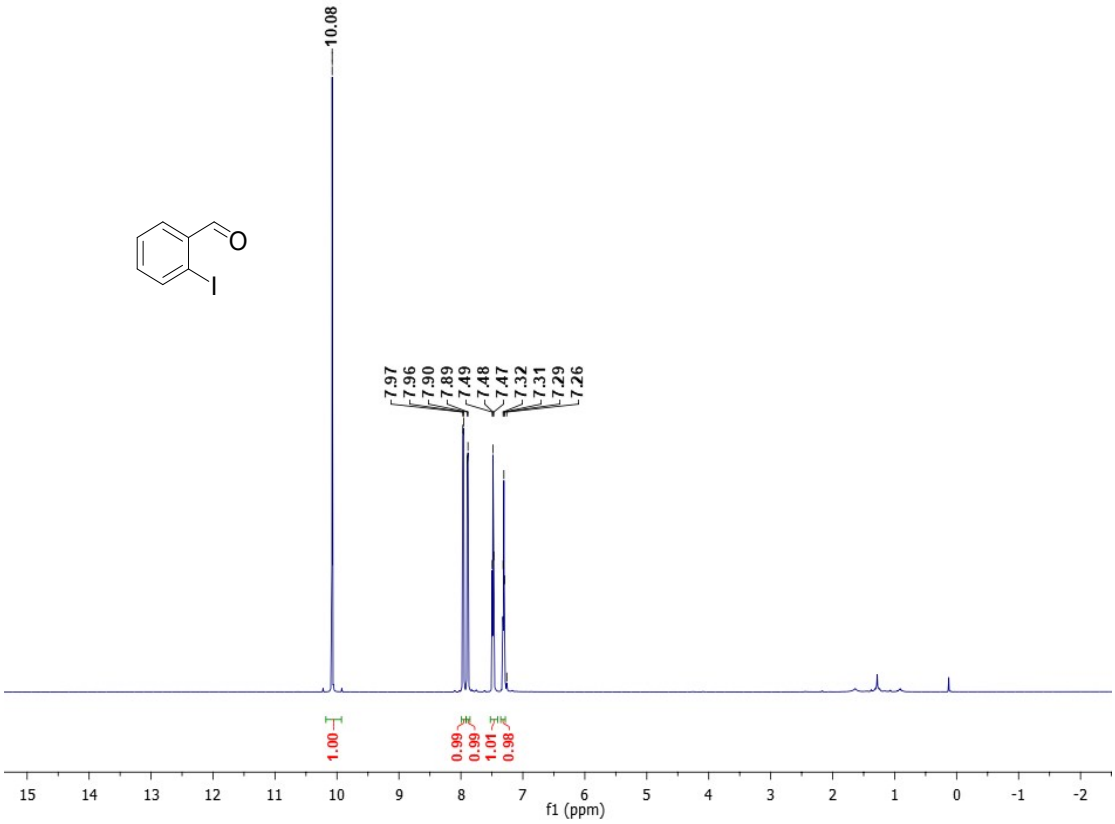


Figure S17: ^{1}H NMR spectrum of **2-iodobenzaldehyde** in $CDCl_3$.

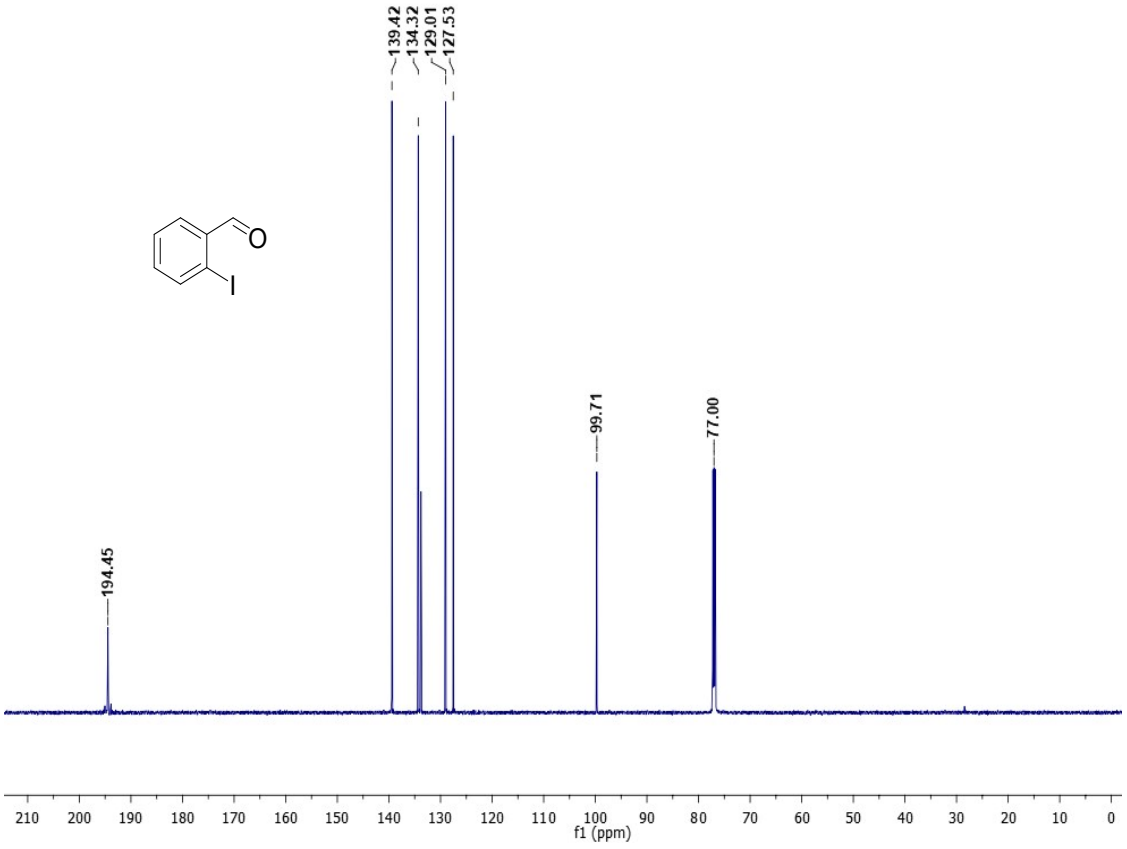


Figure S18: $^{13}C\{^{1}H\}$ NMR spectrum of **2-iodobenzaldehyde** in $CDCl_3$.

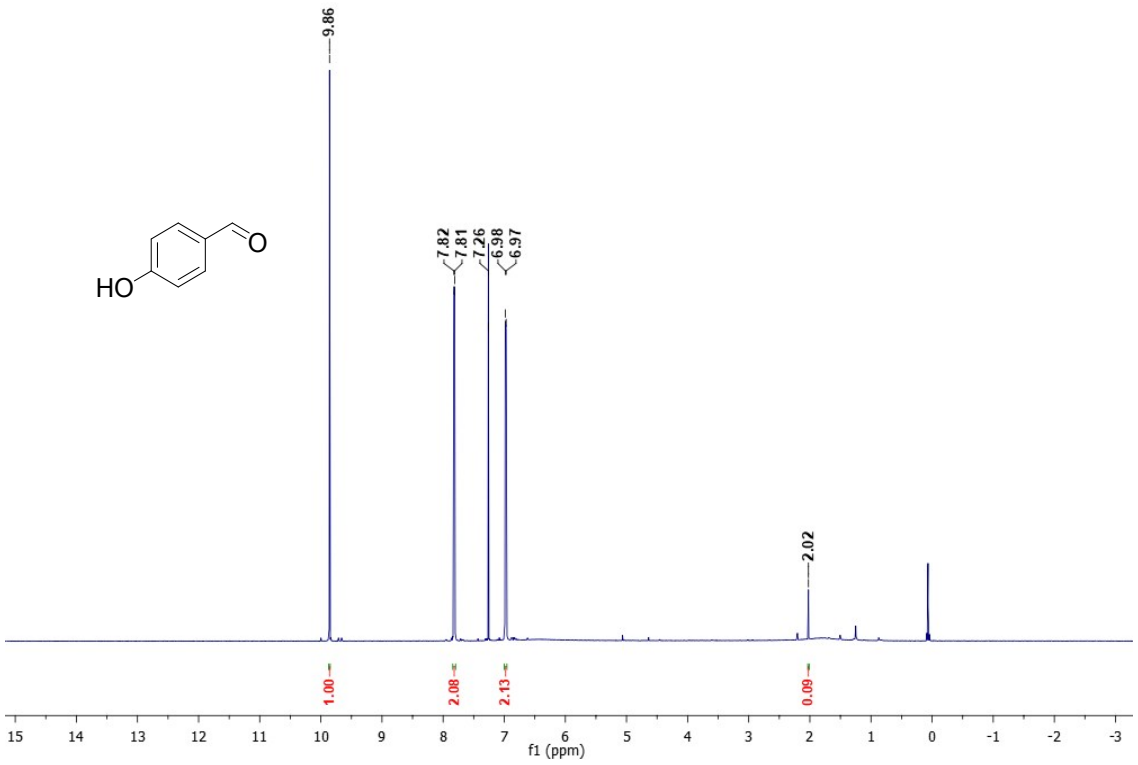


Figure S19: ^{1}H NMR spectrum for **4-hydroxybenzaldehyde** in $CDCl_3$.

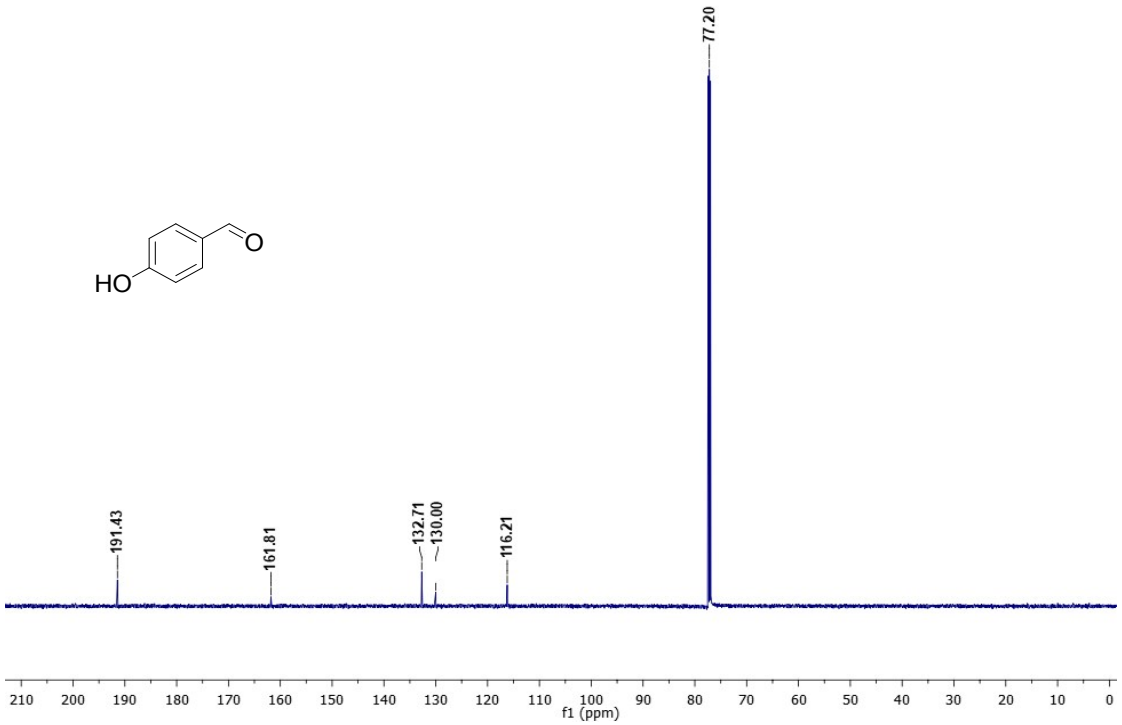


Figure S20: $^{13}C\{^{1}H\}$ NMR spectrum for **4-hydroxybenzaldehyde** in $CDCl_3$.

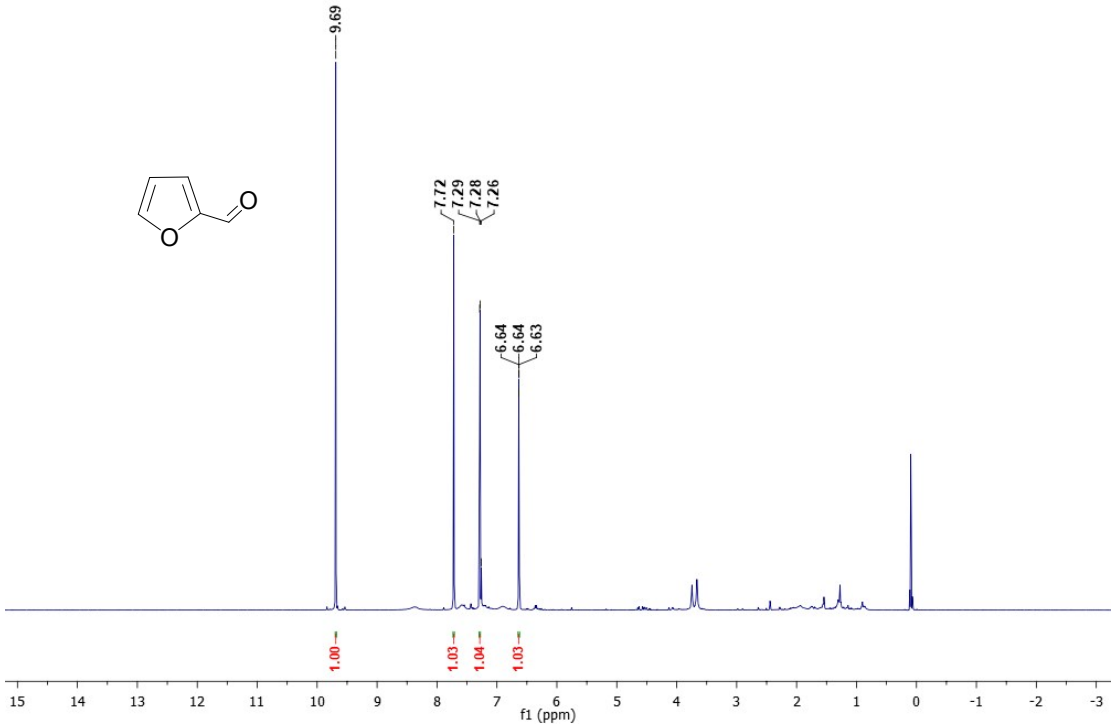


Figure S21: ^{1}H NMR spectrum for **Furfural** in $CDCl_3$.

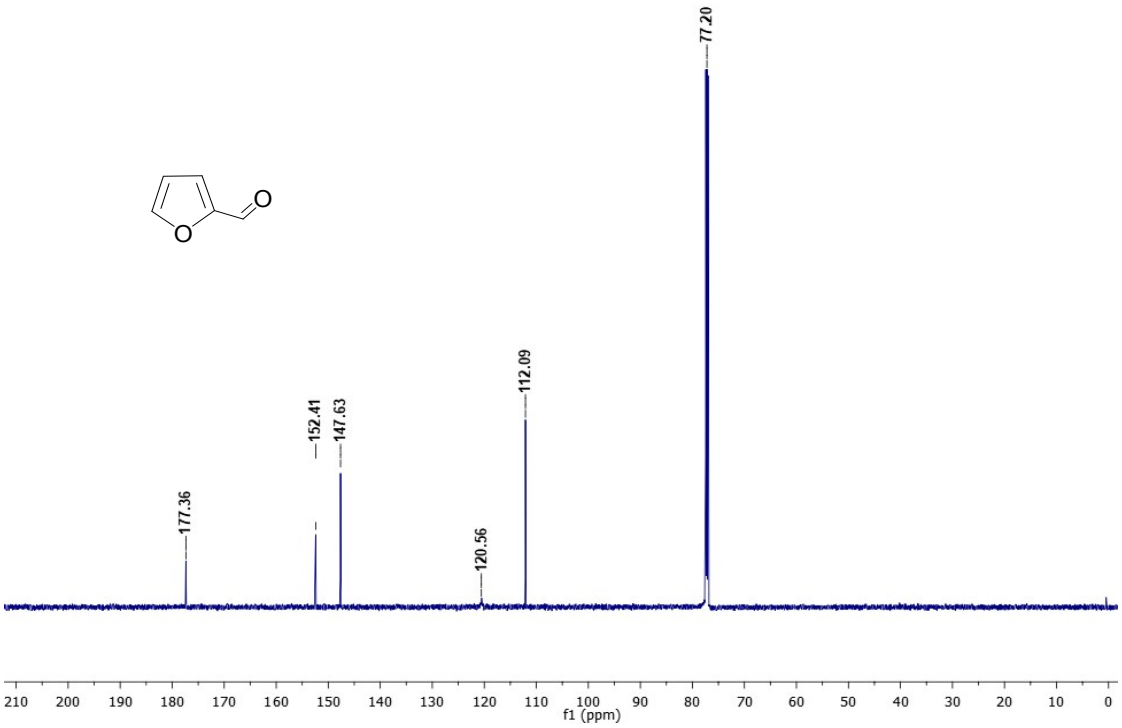


Figure S22: $^{13}C\{^{1}H\}$ NMR spectrum for **Furfural** in $CDCl_3$.

VI. MALDI-MS spectral data for 2-pyridinemethanol

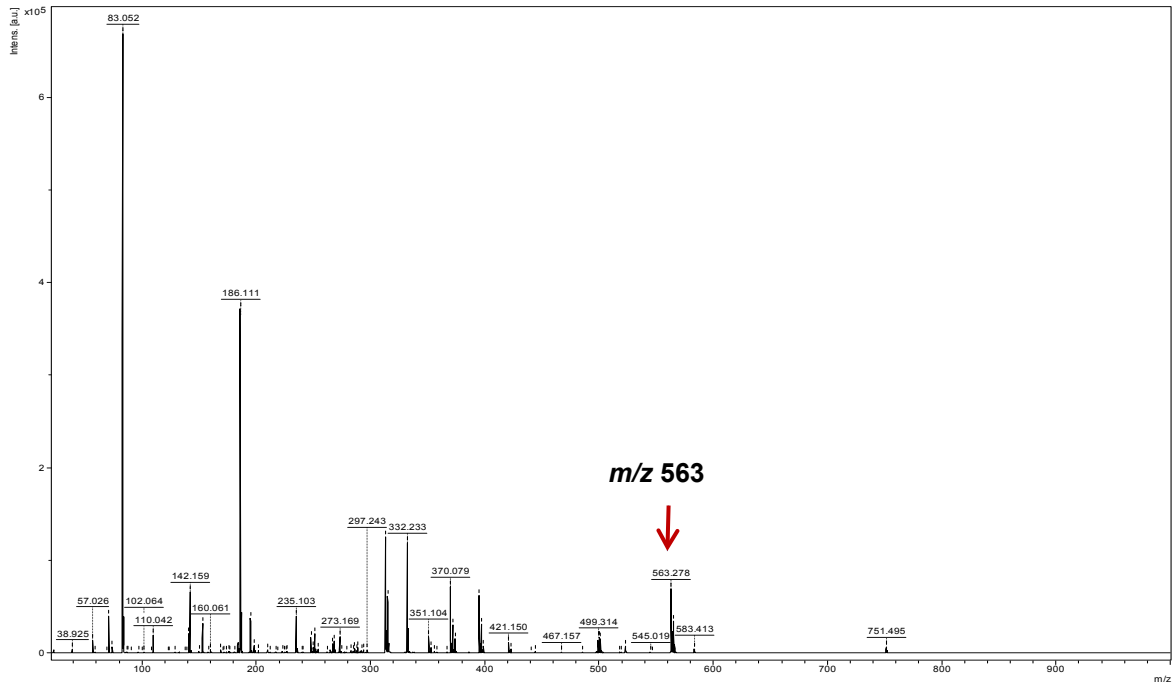


Figure S23: MALDI-MS spectrum of the oxidation reaction employing pyridinemethanol as substrate.

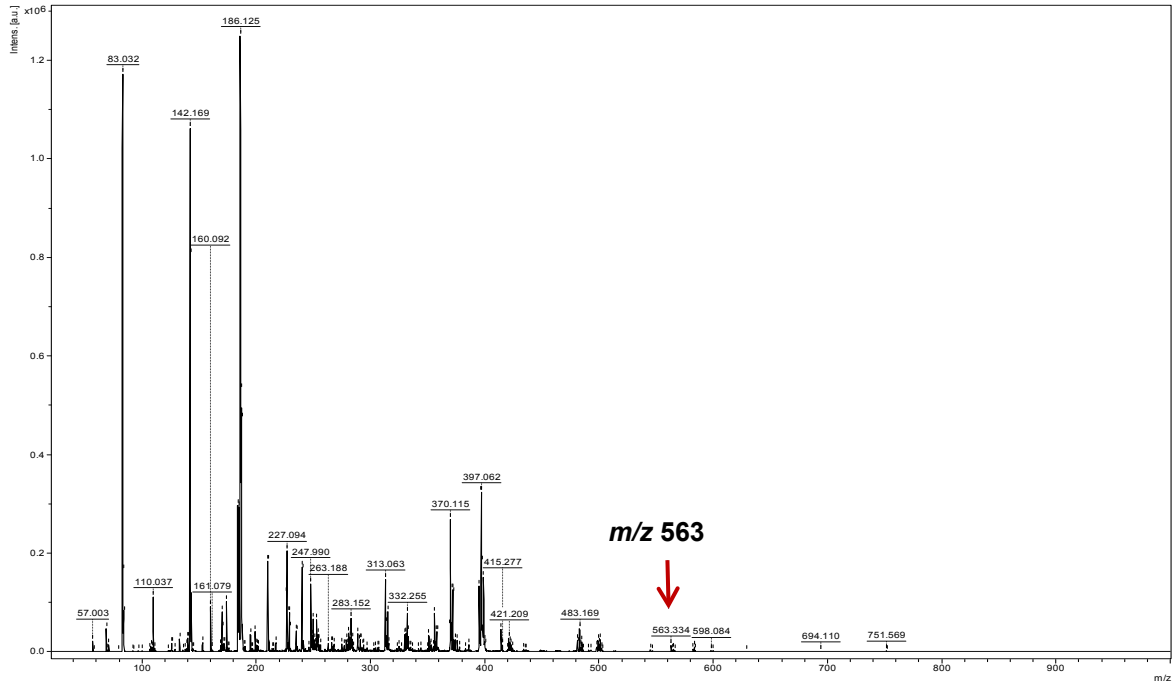


Figure S24: MALDI-MS spectrum of the reaction mixture in the absence of TEMPO, NMI and ligand **L1**.

VII. References

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